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(54) **COATINGS FOR HIGH TEMPERATURE COMPONENTS**

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(58) **Field of Classification Search**

None
See application file for complete search history.

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(57) **ABSTRACT**

A method for forming a coating on a substrate is provided. To an assembly **10** including a substrate **12**, a porous matrix **14** on the substrate **12**, and an impregnating material **16** on or within the porous matrix **14**, the method includes applying an amount of energy **18** from an energy source **20** effective to melt the impregnating material **16** and a portion of the substrate. In this way, the impregnating material **16** impregnates the porous matrix **14**. The method further includes cooling the assembly **10** to provide a coating **26** comprising the porous matrix **14** integrated with the substrate **12**.

18 Claims, 2 Drawing Sheets

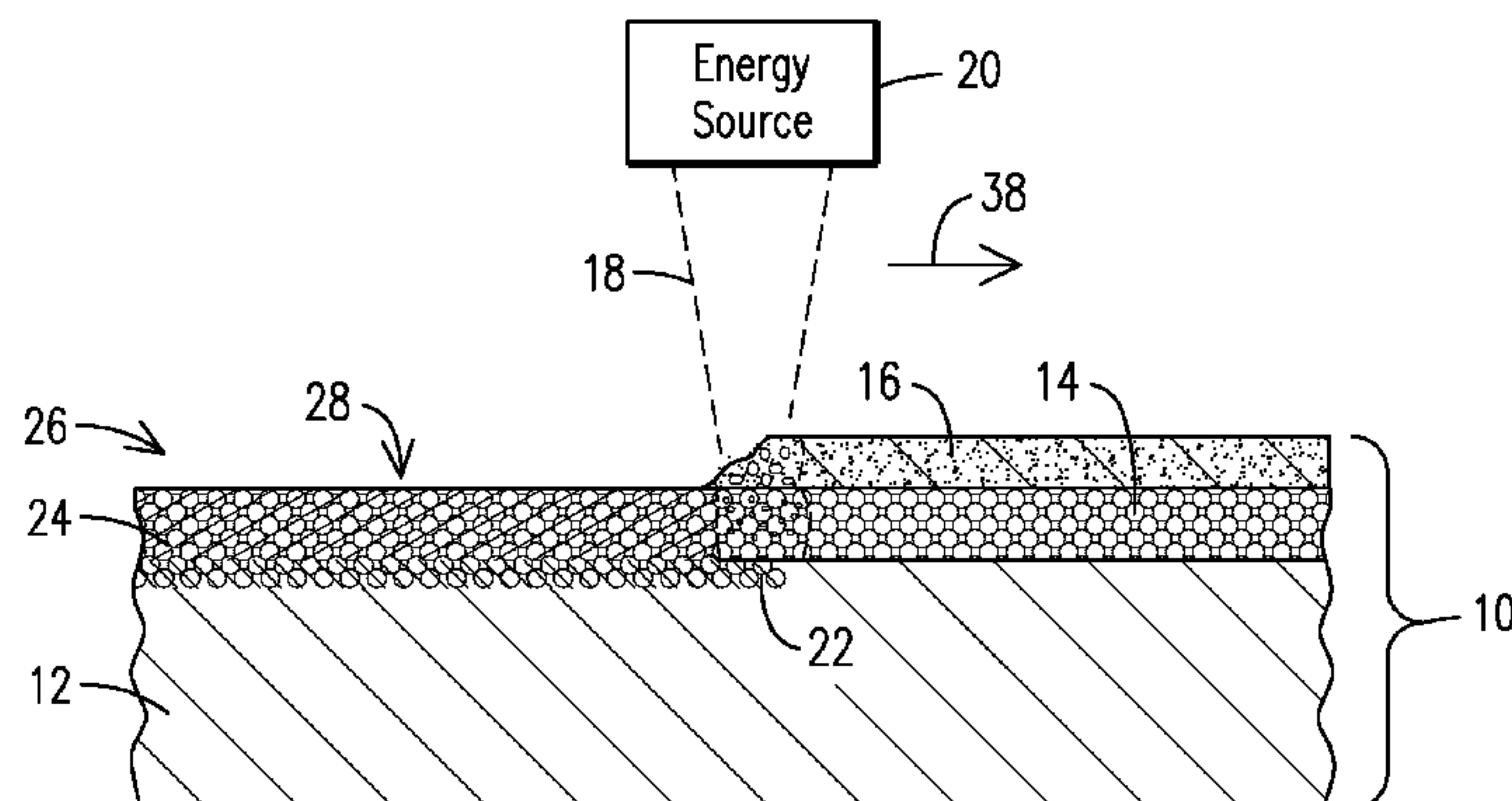


FIG. 1

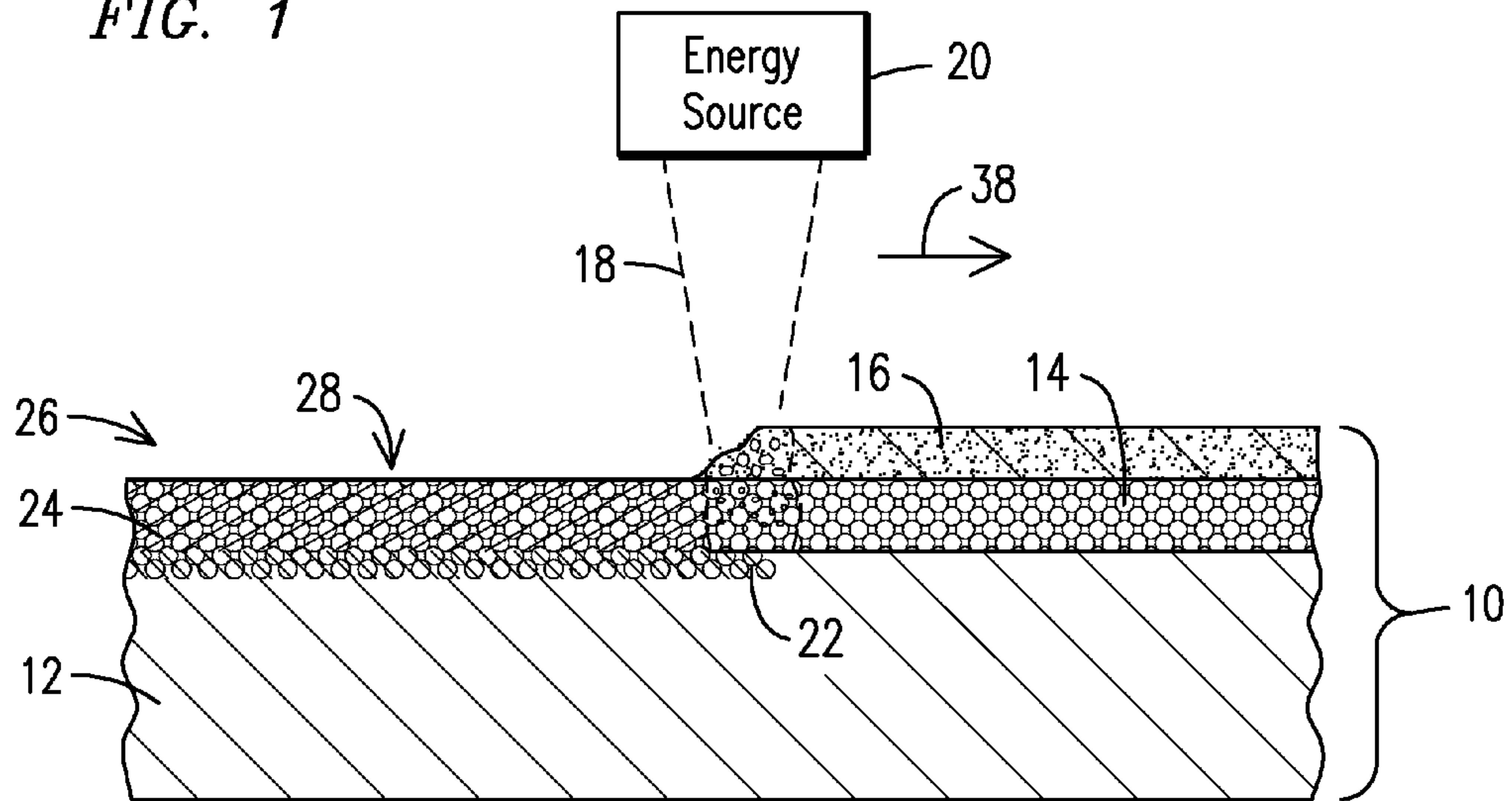
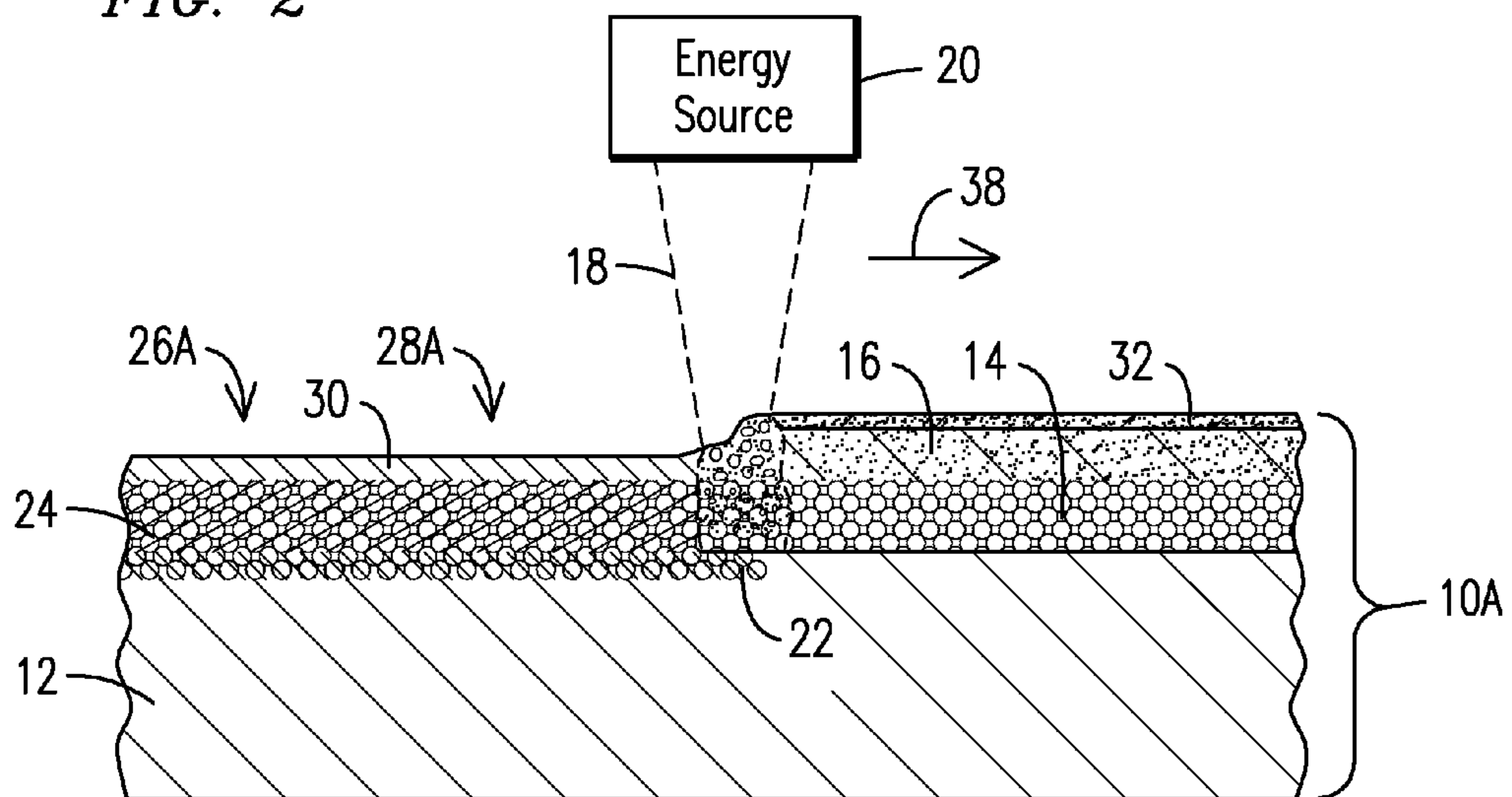
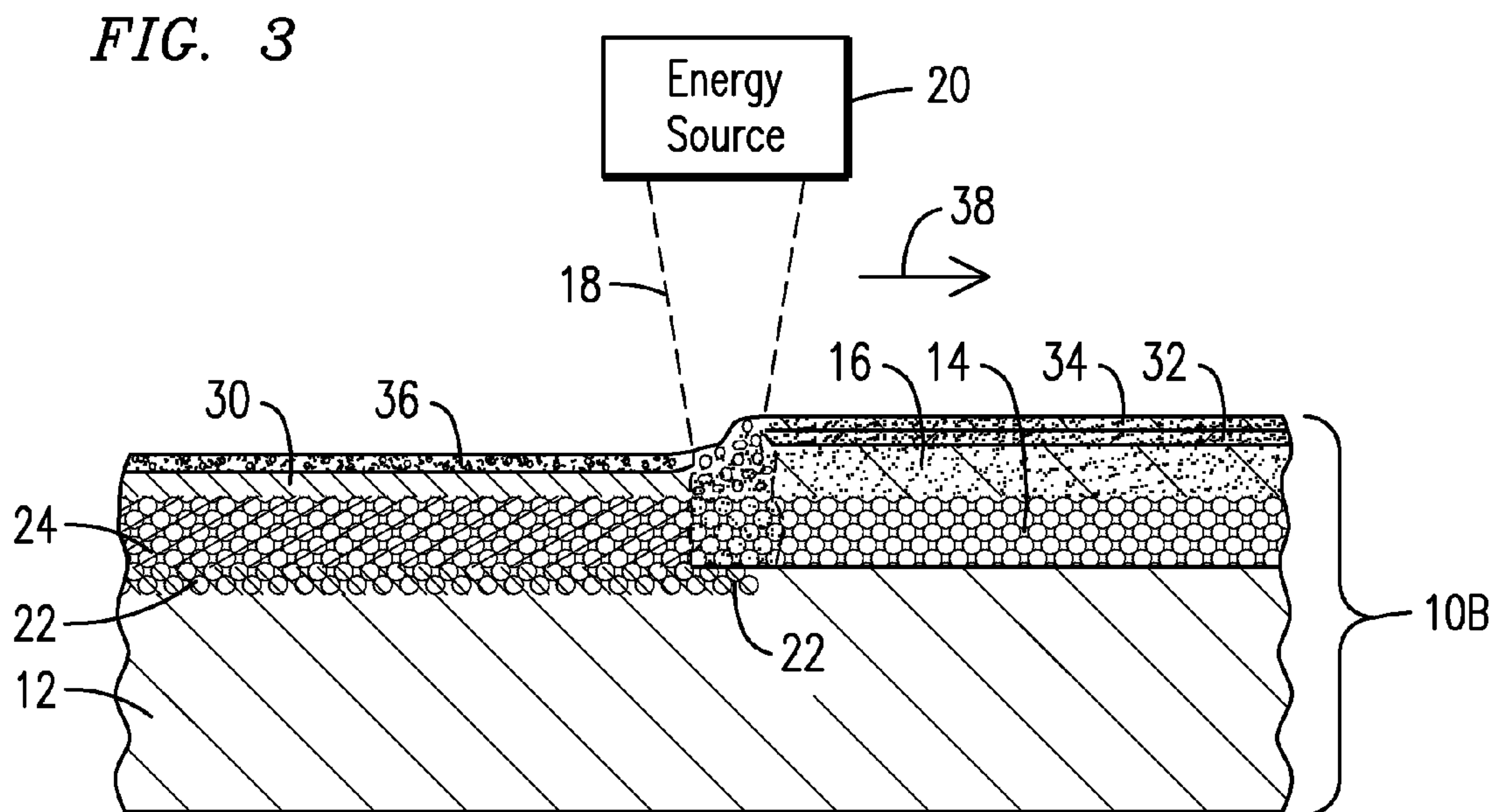


FIG. 2





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COATINGS FOR HIGH TEMPERATURE COMPONENTS

FIELD OF THE INVENTION

This invention relates generally to materials enhancement, and more particularly to processes for providing a strengthening and protective coating on a substrate and to the resulting structures.

BACKGROUND OF THE INVENTION

Structural repair of gas turbine or other superalloy components is commonly recognized as replacing damaged material with matching alloy material and achieving properties, such as strength, that are close to the original manufacture component specifications (e.g., at least seventy percent ultimate tensile strength of the original specification). For example, it is preferable to perform structural repairs on turbine blades that have experienced surface cracks, so that risk of further cracking is reduced, and the blades are restored to original material structural and dimensional specifications. These blades and vanes for land-based power generation and for aerospace applications are typically formed from superalloy materials. The term "superalloy" is used herein as it is commonly used in the art to refer to a highly corrosion-resistant and oxidation-resistant alloy that exhibits excellent mechanical strength and resistance to creep even at high temperatures. Superalloys typically include a high nickel or cobalt content.

Since the development of superalloy materials, various strategies have been employed to provide mechanical strength to the materials to improve the lifetimes thereof during new manufacture or repair. Some elements can provide strength in solid solution. Examples include Co, Cr, Fe, Mo, W, Ta, and Re. Carbide precipitations can also add to strength. Elements forming carbides include W, Ta, Ti, Mo, Nb, Hf and Cr. Most particularly, superalloy materials may be strengthened through the formation of a precipitate phase known as gamma prime. This phase has the basic composition $Ni_3(Al,Ti)$. If properly sized and of sufficient volume fraction, this phase offers significant strengthening—most particularly to nickel based superalloys. Some superalloys are also strengthened by another precipitate known as gamma double prime. This precipitate is of the composition Ni_3Nb , and is important for strengthening some nickel and nickel/iron-based superalloys. Gamma prime phases have an ordered crystalline lattice, which aids in providing added strength to the material. In addition, single crystal solidification techniques have been developed for superalloys that enable grain boundaries to be entirely eliminated from a casting, as well as increase the volume fraction of the γ' precipitates. Alternatively, superalloys may be directionally solidified so as to include only longitudinally directed grains for added strength.

Oxide dispersion strengthened (ODS) alloys are also attractive for high temperature use because of their high temperature strength and oxidation resistance. However, such alloys have found limited application in practice because of the difficulties in manufacture including limited formability (especially of complex shapes), limited machinability, weldability, fabricability or repairability. The potential application of such alloys as a layer or coating is even more challenging due to problems with insufficient bonding of ODS materials to substrates.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention is explained in the following description in view of the drawings that show:

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FIG. 1 illustrates the formation of a coating comprising an impregnated porous matrix in accordance with an aspect of the present invention;

FIG. 2 illustrates the addition of a metal oxide powder to the materials of FIG. 1 to form an outer metal oxide layer on the impregnated porous matrix in accordance with an aspect of the present invention.

FIG. 3 illustrates the inclusion of a powdered flux to the metal oxide powder to form a slag layer in accordance with an aspect of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

The present inventors have developed a novel processes for forming a coating comprising a strengthening and protective high temperature network on a substrate. In certain aspects, the processes described herein place a porous matrix comprising a high temperature material, such as a metal oxide, ceramic, or carbon material, on a substrate and thereafter melt an impregnating material on or within the porous matrix to impregnate the porous matrix with the material. The porous matrix does not substantially melt during the process. A portion of substrate, however, is also melted. The material impregnated into the porous matrix is compatible with the substrate. Thus, when the substrate and molten material cools, the porous matrix is fused to the substrate. In this way, a coating comprising a strengthening and protective high temperature network may be formed on the substrate. In an embodiment, a high temperature material such as a metal oxide material is also disposed on the impregnating material or porous matrix, melted, and cooled to form an outer protective layer that integrates with the non-melted porous matrix to expand the strengthening and protective high temperature material network.

Referring to FIG. 1, there is shown an assembly 10 comprising a substrate 12, a porous matrix 14 disposed on the substrate 12, and an impregnating material 16 disposed on or within the porous matrix 14. In an embodiment, the impregnating material 16 is selected so as to be compatible with the substrate 12 upon melting of the impregnating material 16. The porous matrix 14 may be placed on top of the substrate 12 so as to substantially cover a top surface of the substrate 12 in an area to be joined or repaired, or on a desired area in a new manufacture. The impregnating material 16 may then be pre-placed or otherwise fed onto a surface of the porous matrix 14, for example, as a homogeneous layer. Alternatively, the porous matrix 14 may be provided as a pre-form with the impregnating material 16 already placed thereon, which is placed on the substrate 12.

To form a high temperature protective coating, energy 18 is applied to the assembly 10 from a suitable energy source 20 in an amount effective to melt the impregnating material 16 and to melt a portion 22 of the substrate 12. The energy 18, however, does not substantially melt the porous matrix 14 such that the majority (e.g., >50% by volume) of the structural integrity of the porous matrix 14 remains after the application of the energy 18. In this way, the molten impregnating material 16 flows into and impregnates porous matrix 14 to form impregnated porous matrix 24, which remains substantially self-supporting following energy application. It is contemplated that some of the molten material 16 may also diffuse into the melted substrate (portion 22), but it is at least desirable that the molten material 16 interfaces with the melted substrate portion 22.

During the processes described herein, the melted portion 22 of the substrate 12 may have a desired depth such as from

0.05 to 1.0 mm. Upon cooling and solidification, the porous matrix **14** may become integrated with the substrate **12** via the material **16** within the impregnated porous matrix **24** interfacing and solidifying with the molten portion **22** of the substrate, such as by metallurgical bonding or the like. Advantageously, this anchoring may eliminate coating spallation that is typical of conventionally applied coatings on superalloys, for example. Upon solidification, a protective coating **26** comprising a high temperature network **28** is thus formed on the substrate **12**.

For the processes described herein, the substrate **12** may comprise any material with which would benefit from the processes described herein. In certain embodiments, the substrate **12** comprises a superalloy material. As noted above, the term "superalloy" is used herein as it is commonly used in the art to refer to a highly corrosion-resistant and oxidation-resistant alloy that exhibits excellent mechanical strength and resistance to creep even at high temperatures. Exemplary superalloys include, but are not limited to alloys sold under the trademarks and brand names Hastelloy, Inconel alloys (e.g. IN 738, IN 792, IN 939), Rene alloys (e.g. Rene N5, Rene 41, Rene 80, Rene 108, Rene 142, Rene 220), Haynes alloys, Mar M, CM 247, CM 247 LC, C263, 718, X-750, ECY 768, 262, X45, PWA 1483 and CMSX (e.g. CMSX-4) single crystal alloys, GTD 111, GTD 222, MGA 1400, MGA 2400, PSM 116, CMSX-8, CMSX-10, PWA 1484, IN 713C, Mar-M-200, PWA 1480, IN 100, IN 700, Udimet 600, Udimet 500 and titanium aluminide.

Alternatively, the substrate **12** may comprise a titanium aluminide material, a cermet material, or a ceramic matrix composite or a monolithic ceramic comprising one or more of alumina, zirconia, silica, silicon carbide, silicon nitride, aluminum nitride, silicon oxynitride, silicon carbonitride, mullite, cordierite, beta spodumene, aluminum titanate, strontium aluminum silicates, lithium aluminum silicates, and combinations thereof.

The porous matrix **14** includes a plurality of pores, such as micropores, therein and is formed at least in part from a material that has a higher melting temperature than at least the impregnating material **16**. In this way, the porous matrix **14** may substantially retain its structural integrity even upon application of the energy **18** to melt the impregnating material **16**. The porous matrix **14** may be in the form of a filter, a sponge, a honeycomb, or a foam material, for example.

In one embodiment, the porous matrix **14** may comprise a metal oxide selected from the group consisting of alumina, antimony (III) oxide, boria, chromium oxide, cobalt oxide, copper oxide, europium oxide, iron oxide, nickel oxide, silica, tin oxide, titanium oxide, yttrium oxide, zinc oxide, zirconium oxide (zirconia), and combinations thereof. In a particular embodiment, the porous matrix **14** comprises zirconium oxide, which is particularly advantageous as the zirconia has a higher melting point than most superalloy materials. For example, the porous matrix **14** may comprise a zirconia material such as those commercially available from Foseco or Sigma Aldrich, for example. STELEX ZR zirconium oxide filters are commercially available from Foseco in various sizes and have a porosity of from 10-20 ppi (average number of pores per linear inch).

In another embodiment, the porous matrix **14** may instead or further comprise a titanium aluminide material, a cermet material, or a ceramic material. The ceramic material may comprise, for example, alumina, zirconia, silica, silicon carbide, silicon nitride, aluminum nitride, silicon oxynitride, silicon carbonitride, mullite, cordierite, beta spodumene, aluminum titanate, a strontium aluminum silicate, a lithium

aluminum silicate, and combinations thereof. Ceramic honeycomb is available for use herein, for example, as Versagrid™ from Applied Ceramics Inc. in fine cell patterns up to 400 cells per square inch and of various materials such as alumina, silica, and Mullite.

In still another embodiment, the porous matrix **14** may instead or further include a carbon material to add structural integrity to the final coating **26**. The carbon material may also have a higher melting temperature than the material(s) melted by energy **18**. In addition, the carbon material may be formed from a member selected from the group consisting of a fullerene structure, a carbon yarn, a carbon fiber, a carbon fabric, a carbon honeycomb, a carbon nanobud, a graphene structure, a diamond-like material (e.g. a DLC-coated material), and combinations thereof. By way of example, carbon fiber fabrics are commercially available from Protech Composites and a carbon honeycomb is commercially available from Fiber Glast Nomex.

The impregnating material **16** is selected so as to be compatible with the substrate **12** such that when the material **16** is melted by the energy **18**, the molten material impregnates the porous matrix **14** and integrates with the molten substrate portion **22** to anchor the matrix **14** to the substrate **12**. In an embodiment, the impregnating material **16** comprises a superalloy material as described herein that is compatible with the substrate **12**. In another embodiment, the material **16** comprises a titanium aluminide material, a cermet material, or a ceramic material as described herein that is compatible with the substrate **12**. The impregnating material **16** may have any suitable size range for the intended application. In an embodiment, the material **16** has a particle size of from 5 to 100 μm.

In an embodiment, the amount of impregnating material **16** is carefully selected so as to impregnate the porous matrix **14** without flooding the porous matrix **14** or spilling over its edges. In certain embodiments, the amount of material **16** is selected to substantially fill the porous matrix **14**. For example, in an embodiment, at least 90% of the volume may be filled with molten impregnating material **16**. In certain embodiments, the amount of material **16** is selected such that upon melting the material **16** does not completely fill an internal volume of the porous matrix **14** and an amount of porosity remains in the porous matrix **14** after the cooling. This may, in fact, be desirable in certain applications as the remaining porosity could improve thermal properties of the substrate **12**. For one, existing gases within the pores or void pockets may prevent additional heat from entering into the porous matrix **14**, thereby acting as good thermal barriers and providing added thermal protection to the substrate **12**.

In another embodiment, the porous matrix **14** has a non-uniform porosity therethrough such as along a length of the porous matrix **14**. Advantageously, a non-uniform porous matrix may enhance travel of the melt through the matrix **14** and/or aid in maintaining an amount of porosity in the matrix **14** even after impregnation as described above.

Now referring to FIG. 2, there is shown another embodiment wherein the high temperature network **28** of FIG. 1 may be expanded by the addition of an outer protective high temperature layer **30**. In an embodiment, the outer protective layer **30** is provided by adding a high temperature material, e.g., a metal oxide powder or a ceramic powder, on the assembly as described above with respect to FIG. 1. In particular, referring to FIG. 2, there is provided an assembly **10A** comprising a substrate **12**, a porous matrix **14** disposed on the substrate **12**, an impregnating material **16** disposed on or within the porous matrix **14**, and a high temperature material **32** on the material **16**. The high temperature mate-

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rial **32** may be pre-placed or otherwise fed onto the material **16** as layer of uniform or variable thickness. Alternatively, a porous matrix **14** with the impregnating material **16** already placed thereon and/or therein along with the high temperature material **32** may be provided as a pre-form and placed on the substrate **12**.

The high temperature material **32** may comprise any suitable material which will provide mechanical strength or high temperature resistance properties to the coating. For example, the material **32** may comprise a metal oxide material, a titanium aluminide material, a cermet material, a ceramic material, and combinations thereof. When a metal oxide material is selected, the metal oxide may comprise a member selected from the group consisting of alumina, antimony (III) oxide, boria, chromium oxide, cobalt oxide, copper oxide, europium oxide, iron oxide, nickel oxide, silica, tin oxide, titanium oxide, yttrium oxide, zinc oxide, zirconium oxide (zirconia), and combinations thereof. In a particular embodiment, the porous matrix **14** comprises zirconium oxide. Alternatively, the material **32** may comprise a titanium aluminide, a cermet material or a ceramic material as was described herein with respect to the porous matrix **14**. The high temperature material **32** may be provided in the form of a powder, and in this form may have an average particle size of from 50 to 150 microns.

When subjected to the energy from the energy source **20**, the material **32** is provided in a sufficiently thin layer such that it and the material **16** will be melted without substantially melting the porous matrix **14**. Upon solidification, the outer layer **30** will form as the outermost layer on the substrate **12**. Moreover, since the material **16** flows into and impregnates the porous matrix **14**, the molten material **32** may contact the porous matrix **14**, solidify, and provide a coating **26A** comprising a strengthening and protective high temperature network **28A** on the substrate **12** as shown in FIG. **2**. In certain embodiments, both the porous matrix **14** and material **32** are of the same composition, such as the same metal oxide composition, to form a metallurgically compatible or otherwise compatible network **28A**. In an embodiment, the layer **30** has a final thickness of from 10 microns to 1 mm.

The processes described herein may take place under appropriate flux conditions to shield the molten material(s) from atmospheric oxygen. In certain embodiments, the processes described herein may be performed in a vacuum or in the presence of a flowing stream of argon or other inert gas (over the melt pool which shields the melt pool from atmospheric oxygen).

In other embodiments, a flux powder of a size and composition as described in U.S. Published Patent Application No. 2013/0136868 (the entirety of which is hereby incorporated by reference herein) may be introduced to the process at a suitable point and location to similarly shield the melt pool from atmospheric oxygen. For example, as shown in FIG. **3**, a layer of powdered flux material **34** may also be provided to the components of assembly **10A** (FIG. **2**) to form assembly **10B**. The flux material **34** may be pre-placed or fed onto the material **32** or may otherwise be mixed with the material **32** or impregnating material **16**. For purposes of illustration, the layer of flux material **34** is shown as a distinct layer from the layer of material **32** and material **16**. As the energy **18** melts materials **16**, **32**, and **34** and the molten material solidifies, a slag layer **36** forms on the outside of the weld in addition to the components already described herein.

The slag layer or slag **36** may be removed using any suitable method known in the art, which will leave the

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coating **26A** and network **28A** as was shown in FIG. **2**. It is appreciated that the slag **36** is typically a solid layer that is substantially brittle. In certain embodiments, the slag **36** may be broken by mechanical methods, such as by cracking the slag **36** with a blunt object or vibratory tool, and sweeping away the slag **36** from the cladding. In other embodiments, the slag **36** (once formed) may be of such characteristic to self-detach upon cooling.

The use of a flux powder has a plurality of advantages associated therewith including the formation of a layer of slag at a top surface of the melt pool which aids in carrying away contaminants from the melt pool and in itself insulating and controlling heat dissipation, thereby influencing weld pool shape and stresses.

In the processes described herein, the energy source **20** may be any suitable energy source effective to melt the desired materials, e.g., materials **16** plus **32**, **34** (if present) and the portion **22** of the substrate **12**, but without substantially melting the porous matrix **14** as described above. In an embodiment, the energy source **20** is a laser energy source, although the processes described herein are not so limited. In other embodiments, the energy source **20** may comprise an arc plasma energy source, electron beam or ion beam, for example.

It is appreciated that the skilled artisan would be able to identify the parameters and understand in what manner to vary the parameters so as to provide the intended result such as melting the desired material(s) onto the substrate as well as melt the substrate at a desired rate without substantially melting the porous matrix **14**. Such parameters may include but are not limited to power density, pulse duration, pulse intervals, frequency, the number of energy sources, use of hybrid (e.g. plasma and laser) sources, spacing between source and substrate, and the like. In the processes, it is appreciated the energy source **20** may comprise one or more energy sources that may be traversed with respect to the substrate **12** in a direction of the deposit **38** (FIGS. **1-3**). Alternatively, the substrate **12** may be traversed with respect to the energy source **20** in the direction of the deposit **38**.

In accordance with another aspect, the processes described herein may be further enhanced by the addition of one or more components to any of the materials to be melted (e.g., material **16** and/or material **32**), or the additional components may be added layers as distinct layers prior to melting on the substrate **12**.

For example, in an embodiment, fluorides such as calcium fluoride could be included for improved fluidity of the molten material during processing.

In another embodiment, carbonates such as barium carbonate or calcium carbonate may be added for further shielding of the melt pool from atmospheric oxygen.

In another embodiment, various other oxides such as calcium oxide, magnesium oxide, or manganese oxide may be added to help eliminate solidification cracking.

In another embodiment, alloying elements including nickel, cobalt, aluminum and/or chromium may be added to improve surface oxidation resistance.

In another embodiment, alumina or further alumina may be added to establish still another coating barrier for corrosion, erosion and oxidation resistance.

The processes described herein may be useful for original equipment manufacturing or for prototyping of parts. Furthermore, the processes described herein may be used for component repair applications on components such as a gas turbine blade or vane that has been removed from service for refurbishing.

While various embodiments of the present invention have been shown and described herein, it will be obvious that such embodiments are provided by way of example only. Numerous variations, changes and substitutions may be made without departing from the invention herein. Accordingly, it is intended that the invention be limited only by the spirit and scope of the appended claims.

What we claim is:

1. A method of coating a substrate comprising: to an assembly comprising a substrate, a porous matrix on the substrate, and an impregnating material on or within the porous matrix, applying an amount of energy from an energy source effective to melt the impregnating material and a portion of the substrate such that the impregnating material impregnates the porous matrix; and cooling the assembly to provide a coating comprising the impregnated porous matrix integrated with the substrate; wherein the porous matrix comprises a material having a higher melting temperature than that of the impregnating material.
2. The method of claim 1, wherein the substrate and the impregnating material comprise a composition selected from the group consisting of a superalloy material, titanium aluminide, a cermet material, and a ceramic material.
3. The method of claim 1, further comprising forming the assembly via: disposing the porous matrix on the substrate; and disposing the impregnating material on or within the porous matrix.
4. The method of claim 1, wherein the porous matrix comprises a metal oxide selected from the group consisting of alumina, antimony(III) oxide, boria, chromium oxide, cobalt oxide, copper oxide, europium oxide, iron oxide, nickel oxide, silica, tin oxide, titanium oxide, yttrium oxide, zinc oxide, zirconium oxide, and combinations thereof.
5. The method of claim 1, wherein the porous matrix comprises a ceramic material selected from the group consisting of alumina, zirconia, silica, silicon carbide, silicon nitride, aluminum nitride, silicon oxynitride, silicon carbonitride, mullite, cordierite, beta spodumene, aluminum titanate, a strontium aluminum silicate, a lithium aluminum silicate, and combinations thereof.
6. The method of claim 1, wherein the porous matrix further comprises a material selected from the group consisting of a fullerene structure, a carbon yarn, a carbon fiber, a carbon nanobud, a graphene structure, a diamond-like carbon coated material, a carbon honeycomb, a carbon fabric, and combinations thereof.
7. The method of claim 1, further comprising: adding a high temperature material to a top of the assembly, wherein the high temperature material is selected from the group consisting of a metal oxide material, titanium aluminide, a cermet material, and a ceramic material; melting the high temperature material during the applying an amount of energy from an energy source; and cooling the molten high temperature material to form an outer layer therefrom on the coating which is integrated with the porous matrix.
8. The method of claim 7, wherein the high temperature material comprises a metal oxide material selected from the group consisting of alumina, antimony(III) oxide, boria, chromium oxide, cobalt oxide, copper oxide, europium

oxide, iron oxide, nickel oxide, silica, tin oxide, titanium oxide, yttrium oxide, zinc oxide, zirconium oxide, and combinations thereof.

9. The method of claim 7, wherein a portion of the impregnating material is melted during the applying an amount of energy from an energy source.

10. The method of claim 7, further comprising:

adding a flux powder to the assembly; and

melting the flux powder during the applying an amount of energy from an energy source to form a removable slag layer on the outer layer of the coating.

11. The method of claim 1, wherein the porous matrix has a non-uniform porosity therethrough.

12. The method of claim 1, wherein an amount of porosity remains in the porous matrix after the cooling.

13. A method for forming a coating on a substrate comprising:

to an assembly comprising a substrate, a porous matrix on the substrate, an impregnating material on or within the porous matrix, and a high temperature material selected from the group consisting of a metal oxide material, titanium aluminide, a cermet material, and a ceramic material on an outer portion of the assembly, applying an amount of energy to the assembly from an energy source,

wherein the amount of energy is effective to melt the high temperature material, the impregnating material, and a portion of the substrate such that the impregnating material impregnates the porous matrix;

wherein the porous matrix comprises a material having a higher melting temperature than that of the impregnating material; and

cooling the assembly to provide a coating comprising a network integrated with the substrate, the network comprising the porous matrix and an outer layer formed from the high temperature material.

14. The method of claim 13, wherein the substrate and the impregnating material comprise a material selected from the group consisting of a superalloy material, titanium aluminide, a cermet material, and a ceramic material.

15. The method of claim 13, further comprising forming the assembly via:

disposing the porous matrix on the substrate;

disposing the impregnating material on or within the porous matrix; and

disposing the high temperature material on the impregnating material.

16. The method of claim 13, wherein the porous matrix and the high temperature material comprise a metal oxide material selected from the group consisting of alumina, antimony(III) oxide, boria, chromium oxide, cobalt oxide, copper oxide, europium oxide, iron oxide, nickel oxide, silica, tin oxide, titanium oxide, yttrium oxide, zinc oxide, zirconium oxide, and combinations thereof.

17. The method of claim 13, wherein the porous matrix and the high temperature material comprise a ceramic material selected from the group consisting of alumina, zirconia, silica, silicon carbide, silicon nitride, aluminum nitride, silicon oxynitride, silicon carbonitride, mullite, cordierite, beta spodumene, aluminum titanate, a strontium aluminum silicate, a lithium aluminum silicate, and combinations thereof.

18. The method of claim 13, wherein an amount of porosity remains in the porous matrix after the cooling.